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(12) **Patent:**

(11) **CA 914144**

(54) MATERIALS FOR EXTREME PRESSURE LUBRICATION

(54)

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ABSTRACT:

CLAIMS: [Show all claims](#)

\*\*\* Note: Data on abstracts and claims is shown in the official language in which it was submitted.

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DONALD O. DETHMERS

MATERIALS FOR EXTREME  
PRESSURE LUBRICATION

ABSTRACT OF THE DISCLOSURE

Metaphosphate monoesters when incorporated into a lubricating oil have been found to be effective as extreme pressure lubricants. A method of extreme pressure lubrication is also disclosed.

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### INTRODUCTION

10 The main function of a lubricating film is to reduce the amount of intimate metallic contact between sliding metal surfaces. Under extreme conditions, most lubricating compositions will break down. These extreme conditions can be high speed, high temperatures, or hard surfaces, such as hard steel. Under these extreme conditions even the best type of known lubricants may break down and consequently there will be an increase in friction between the metallic surfaces and resulting surface damage. The high temperatures and the hard surfaces can cause the lubricant to break down.

Contact without a lubricant or after the lubricant has broken down will cause deformation or welding. Shearing, tearing, or wearing of the engaged surfaces also occur. Localized welding may also be caused. The result is that the metal surfaces become rough and pitted. If the finished product requires smooth surfaces such as car bodies, the unseemly appearance caused by the above conditions makes the metal unusable.

20 To overcome these disadvantages, an extreme pressure additive is used with the lubricating oil or grease. This additive will prevent welding. It will also lower the shear strength and reduce metal tearing and wearing. Under the extreme conditions of temperature, hard surfaces, and high speeds, these additives do not break down.

### OBJECTS

It is an object of this invention to provide an extreme pressure additive that does not break down under the extreme conditions of temperature, hard metal surfaces, and high speeds.

It is a further object of this invention to provide a lubricating composition that contains the above additive.



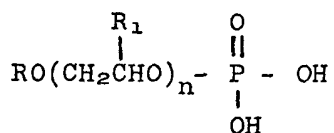
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A further object is to provide a method of lubricating metals under condition of high temperatures and pressures.

Further objects will be apparent to those skilled in the art.

#### THE INVENTION

The extreme pressure additives used in this invention are monoalkyl esters of metaphosphoric acid. These esters are represented by the formula:



where R is an alkyl radical of from 6-22 carbon atoms in chain length,  $\text{R}_1$  is selected from the group consisting of hydrogen and methyl, and n is a small whole number from 0-20.

The above additive is incorporated into a lubricant to make an extreme pressure lubricating composition. The lubricant can be selected from the group consisting of lubricant oils and greases. A wide variety of conventional materials which exhibit lubricant properties may be used for the base lubricating oils or greases. For example, the hydrocarbon oils, synthetic lubricating oils, or mixtures of these in various proportions may be used. The hydrocarbon oils may include mineral oil, vegetable and animal oils such as paraffin bases, naphthene bases or mineral base oils of the residual or distillate type. A particular oil of this type is a paraffin base oil which has been solvent dewaxed and clay percolated. Other preferred lubricants include straight mineral lubricating oils or distillates, bright stock residue and the like.

Synthetic lubricating base oils may also be employed with success. Typical examples are high molecular polyalkylenes, such as polybutene and high molecular weight, high boiling liquid

aliphatic dicarboxylic acid esters. Other useful synthetic oils include polyglycols and polyester lubricants. Sulphur analogues of any of the above-described diesters, polyesters, and polyalkylene ethers may also be usefully employed in formulating the lubricating composition of this invention. Hydrogenated oils are another typical synthetic lubricating oil. Greases derived from any of the above fluid lubricants may be employed as useful extreme pressure lubricants when used in combination with the monoalkyl ester of metaphosphoric acid.

10

The lubricating compositions of the invention may contain other additives such as are conventionally employed in lubricating oils such as detergents, anti-oxidants, viscosity improvers, anti-foam agents, corrosion inhibitors, etc. A typical anti-oxidant is an alkyl-substituted phenol such as 2,6 di-tertiary butyl-4-methyl phenol. A useful class of anti-rust compounds is the alkyl-substituted aliphatic dicarboxylic acids such as alkenyl-succinic acids. A typical anti-foam may be selected from the organic silicone class such as dimethyl silicone.

#### PREPARATION OF THE ESTERS

20

The metaphosphate monoesters used in preparing the lubricant composition of the invention are readily synthesized by using known preparative techniques. Several of these techniques are described in the book Phosphorous and its Compounds by John R. VanWazer, Volume II, Interscience Publishers, Inc., New York, 1961. A preferred method for preparing the monoesters resides in reacting two moles of a starting alcohol with one mole of  $P_2O_5$ . This reaction is normally conducted by slowly adding the  $P_2O_5$  to the alcohol and during the course of addition maintaining the temperature of the reaction at about  $120^\circ C$ . The reaction is

substantially complete over a period from one to five hours. After the reaction described above is complete, water is added to the reaction product to produce by means of hydrolysis the finished monoester used in the practice of the invention.

The above described reaction produces in high yield the monoester of metaphosphoric acid. Due to the poly-functionality of the starting phosphorous pentoxide, minor amounts not exceeding 10% by weight of the diester are produced. The superiority of the lubricating composition is due to the preponderance of the monoester.

10

#### THE STARTING ALCOHOL

The alcohols used to produce the esters of this invention may be selected from either simple monohydric aliphatic alcohols or from monohydric aliphatic alcohols which have been reacted with various quantities of either ethylene or 1, 2-propylene oxides. Illustrative of the aliphatic monohydric alcohols are hexanol, heptanol, octanol, nonanol, decanol-1, decanol-4, dodecanol, tetradecyl alcohol, cetyl alcohol, stearyl alcohol, 2, 6-dimethyl-3-methylolheptane, and many others. The starting alcohols should have from 6-22 carbon atoms in chain length, with from 15-22 carbon atoms being the preferred range.

20

#### PROPORTIONS

The lubricant can make up from 90-99.9% of the lubricating composition. The preferred range is from about 95-98% by weight. The lubricant can be any one of the previously described fluid lubricants and greases. Different results can thus be achieved depending upon the lubricant used.

The monoalkyl ester of metaphosphoric acid can range from 0.1-10% by weight of the lubricating composition. The preferred range is from about 2-5% by weight. Although the straight chain primary alcohol has been found effective in making the ester, the ethoxylated and propoxylated fatty alcohols can also be used and not depart from the invention. Any known means can be used for preparing the ester of metaphosphoric acid.

10 The lubricating composition has also been found effective when mixed with water to form an emulsion. The lubricating composition is prepared in the same way as previously described. Then, the lubricating composition is added to water to form the emulsion. The lubricating composition can range from 1-10% by weight based on the weight of the water. This emulsion has been found useful as a lubricating composition.

#### EXAMPLES

20 In order to better understand the invention the following table was prepared using several different esters. Alcohols of different chain length were used to prepare the ester according to the previously described procedure. One of the alcohols was also ethoxylated to test out this effect. Another additive, polyethoxy octyl phenol, was also tested to show the effect of the additive. After the esters were made using these various alcohols, they were incorporated with a lubricant into a lubricating composition. They were then tested on the Timken and Falex Lubrication Tests.

The lubricating compositions so produced were subjected to the Timken load-bearing tests in the Timken Lubricant Tester in which a hardened steel ring rotating at 800 rpm was flooded by the test lubricating composition at 100°F. while a hardened steel block was pressed against it by a system of weights and levers.



The highest weight which did not produce scoring of the block in ten minutes running time was recorded as the passing load or O.K. load, which is a recognized measure of the load-carrying capacity of a lubricant. Therefore, the higher the weight which can be applied without producing scoring, the better the lubricating composition.

10

The Falex Lubricant Tester consists of a test pin suspended between two jaws. The lubricating composition flows between the test pin and the jaws. These jaws can be tightened around the test pin and thus increase the load. There is a gauge which indicates the increasing load. The maximum load that can be applied without welding the test pin, causing it or the shear pin securing the test pin to break, is the measure of the effectiveness of the lubricating composition. The maximum reading of the Falex Lubricant Tester is 4500 pounds.

The Timken and Falex tests are popular tests known in the art. They are described in Encyclopedia of Chemical Technology, Second Edition, Volume 12, pages 559-564, Interscience Publishers, 1968.

20

The values obtained in accordance with these standard tests using various extreme pressure additives are set forth in Table I.

TABLE I

COMPOSITION NUMBER	THEOR. C NO. OF ALC.	THEOR. EQUIV. WT.	CARRIER	% ESTER	% CARRIER	TEST TIMKEN	RESULTS FALEX NO. 8
1	C7	107	100 sec Paraffin Oil	2%	98%	Insoluble	Insoluble
2	C9	113	100 sec Paraffin Oil	2%	98%	Insoluble	Insoluble
3	C12	132	100 sec Paraffin Oil	2%	98%	70 pounds	3500 pounds
4	C13	138	100 sec Paraffin Oil	2%	98%	80 pounds	4500 pounds
5	C13 + 3 MEO	208	#10 Industrial Oil	3%	55%	70 pounds	3500 pounds
6	C13 + 3 MEO	208	#10 Industrial Oil	3%	55%	70 pounds	4500 pounds
7	C14	152	100 sec Paraffin Oil	2%	98%	-	3750 pounds
8	C16	162	100 sec Paraffin Oil	2%	98%	100 pounds	4500 pounds
9	C17	167	100 sec Paraffin Oil	2%	98%	100 pounds	4500 pounds
10	C18	175	100 sec Paraffin Oil	2%	98%	60 pounds	4500 pounds
11	C20	187	100 sec Paraffin Oil	2%	98%	90 pounds	4250 pounds
12	C21	198	100 sec Paraffin Oil	2%	98%	90 pounds	4000 pounds
13	C22	208	100 sec Paraffin Oil	2%	98%	-	4500 pounds
14	C22	208	#10 Industrial Oil	3%	55%	70 pounds	4500 pounds
15	Octyl Phenol + 4 MEO	-	#10 Industrial Oil	3%	55%	8 pounds	3500 pounds
16	C9 + - MEO	-	#10 Industrial Oil	5% (Emulsion)	41%	80 pounds	-

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As can be seen from the various compositions such as 1, 2, 3 and 4 the chain length of the alcohol can have an effect upon the results. Compositions number 1 and 2 formed insoluble compositions and therefore were not suitable for oil based extreme pressure lubricants. Composition number 4 which had a theoretical number of 13 carbon atoms gave better results than a theoretical number of 12 carbon atoms of alcohol, as can be seen from the table.

10 Example 5 and 6 show that the alcohol of 13 carbon atoms was ethoxylated with three moles of ethylene oxide. As can be seen by composition number 5 this was not as good as composition number 4 which was the alcohol that was not ethoxylated. Composition number 6 was better than composition number 5. The difference was that composition 6 was made using a ratio of alcohol to phosphorous pentoxide of 1.75:1. Composition number 5 was prepared using an alcohol to phosphorous pentoxide of 2:1. This indicates that when we use less than the 2:1 ratio more of the monoalkyl ester is produced than the diester of phosphoric acid, and the resulting monoalkyl ester of phosphorous acid is better for extreme pressure lubrication.

20 Compositions number 13 and 14 were compared using different carriers. Composition number 13 consisted of 98% of a 100 second paraffin oil. Composition number 14 consisted of 55% of Number 10 Industrial Oil. The rest of the total composition was made up of a mixture of oils and greases. As can be seen, composition number 14 gave better results than the corresponding composition number 13.

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Composition number 15 consisted of octyl phenol ethoxylated with four moles of ethylene oxide using 100 second paraffin oil as the carrier. As can be seen from the Timken and Falex Tests, this was not effective as an extreme pressure lubricant.

Composition number 16 consisted of a lubricating composition made using 5% of the additive which had a theoretical number of 9 carbon atoms ethoxylated with four moles of ethylene oxide. The additive was mixed with No. 10 Industrial Oil, other oils and greases as the carrier. Then 5% of this lubricating composition was mixed with water to form an emulsion. The emulsion was tested and showed effective lubrication on the Timken Tester.

#### CONCLUSIONS

The monoalkyl esters of metaphosphoric acid is very effective under extreme conditions as a lubricating composition. The incorporation of these esters into a regular lubricating oil prevents shearing, tearing, welding, and wearing of the metallic surfaces when they come into contact with each other. This is an improvement over the art and is far superior to the known extreme pressure compositions now in use. When this lubricating composition is applied as a thin lubricating film, it is effective in reducing wearing, tearing, shearing, welding and scuffing of the engaging metal surfaces.

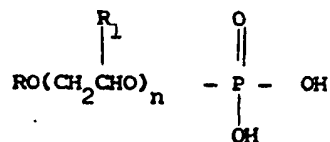
What I claim and wish to protect by Letters and Patents is:

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Claims

1. An extreme pressure lubricating composition for inhibiting welding, tearing, wear, scuffing, and seizure of engaging metals which comprises:

- A. of from 90 to 99.9% by weight of a lubricant selected from the group consisting of fluid lubricants and greases, and
- B. of from 0.1 to 10.0% by weight of a monoalkyl ester of phosphoric acid wherein said ester has the formula:



where R is an alkyl radical of from 7-22 carbon atoms in chain length,  $R_1$  is methyl, and n is a small whole number from 0 to 4 inclusive.

2. The composition of Claim 1 wherein said lubricant is a hydrocarbon lubricating oil.

3. The composition of Claim 1 wherein said alkyl radical is a saturated, straight chain hydrocarbon radical of from 15-22 carbon atoms in chain length and n is 0.

4. The composition of Claim 1 wherein  $R_1$  is the methyl radical, and n ranges from 1-4.

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<sup>5</sup>  
8. An emulsion composition for extreme pressure lubrication consisting of from 90-99% water and from 1-10% of the lubricating composition of Claim 1.

<sup>6</sup>  
7. The composition of Claim 1 wherein said lubricant is a hydrocarbon lubricating oil and consists of 97% by weight of the composition and the monoalkyl ester of phosphoric acid consists of 3% by weight of the composition.

<sup>7</sup>  
8. A method of extreme pressure lubrication to retard welding, tearing, wear, scuffing, and seizure of engaging metal surfaces operating at high pressures or high speeds, which comprises maintaining on such surfaces a thin lubricating film of the lubricating composition of Claim 1.

<sup>8</sup>  
9. A method of extreme pressure lubrication to retard welding, tearing, wear, scuffing, and seizure of engaging metal surfaces operating at high pressures or high speeds, which comprises maintaining on such surfaces a thin lubricating film of the lubricating composition of Claim <sup>5</sup>8.

